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THE EFFECT OF ADDED WATER AND ANTIOXIDANTS ON THE KEEPING QUALITY OF LARD[1]

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Abstract

Moisture alone had little detrimental effect on lard storage life (time at 90° F. to attain a peroxide oxygen value of 10 ml. of 0.002 *N* thiosulphate per gm.) except at the highest levels of incorporation (up to 12.8%), but citric acid, lecithin, and alpha-tocopherol had more stabilizing action in dry lard than in lard containing water. Gum guaiac gave the greatest increase in storage life and citric acid the least. The stabilization varied with the original quality of the lards tested (two wet rendered, two dry rendered, and two composite lards) but no segregation of the results according to wet and dry *rendering* of these lards was possible.

Introduction

The presence of water in fat tends to increase enzymic and microbial changes and may affect oxidative deterioration. In the few investigations of the direct action of water on fat oxidation various results have been reported. With butterfat a small (unstated) concentration of added water had no effect (3), 4% water extended the induction period (9), and 16% water accelerated oxidation in one study (11) but had no effect in another (19). The addition of small amounts of water to dried lard did not change its stability (6). The induction period of vegetable oils was reduced by treatment with boiling water, presumably because natural antioxidants were destroyed (18). In systems containing constituents other than fat, water may act indirectly as an antioxidant by increasing the stabilizing action of water-soluble substances, or by stimulating microbial growth which may produce a condition unfavorable for oxidation of the fat (12, p. 184). In the wet rendering of lard, it is important to exclude tankage water, which carries food for bacteria. Water may destroy the activity of stabilizers for dry fats (12, p. 174), or increase the activity of certain antioxidants (15). Because of the limited information available it was considered of interest to study the effect of moisture on fat oxidation, using as experimental materials Canadian lard and the antioxidants permitted for use in Canada.

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Materials and Methods

To investigate the effect of water on the stability of representative lards produced in Canada, 75-lb. batches of two wet rendered lards (*A* and *B*) and two dry rendered lards (*C* and *D*), free of added antioxidants, were obtained from four plants. These were selected on the basis of a previous survey (14) to secure both poor (*A* and *C*) and fair (*B* and *D*) samples of each of the two types. Two mixtures (*E* and *F*), each containing several wet and dry rendered lards, were also used as test materials.

Preparation of Storage Samples

Preliminary experiments indicated that all the water in 3000 to 3500 gm. lots of commercial lard (about 0.1% moisture) could be removed by heating under vacuum in a boiling water bath, with continuous agitation, for three hours. The lard was considered dry if a 2 gm. sample remained constant in weight when heated in a vacuum oven for two hours at 122° F. (1).

Preliminary work also demonstrated that an emulsion of water in lard containing up to 12% water was stable when chilled and then brought to room temperature, but above this moisture level there was some separation of water. Incorporating water by beating or stirring solid or liquid lard was a lengthy procedure and it was difficult to obtain a uniform mix. Two types of colloid mills were unsuitable for this purpose because the lard was heated too drastically during the milling process. Finally, a Waring Blendor was found to produce a reasonably stable emulsion without excessive temperature rise, when components initially at room temperature were blended for five minutes and then cooled immediately at 25° F. Cooling at lower temperatures caused separation of some of the added water.

The following procedure for preparation of samples was adopted. Melted, dry lard (500 gm.) was weighed into Waring Blendor jars and measured amounts of redistilled water added. The lard was allowed to become semi-solid at room temperature before blending to minimize loss of water by evaporation and to produce a tighter emulsion. The Blendor assembly was placed under a bell jar, the internal atmosphere replaced with tank nitrogen, and the mixture blended for five minutes. The emulsion was immediately transferred to a sealed quart container, and held at 25° F. until needed. The above method was termed Blending Procedure I and was used for all experiments with the individual wet and dry rendered lards. To provide uniformly processed test material, samples containing zero moisture were also given the blending treatment.

With the composite lards the blending procedure was altered (Blending Procedure II). The lard was filtered, before processing, in a Seitz filter (K-2 paper) to remove suspended material that might encourage growth of micro-organisms. All blending was done in a 40° F. constant temperature room. Waring Blendors were run in air rather than in nitrogen, and the blended samples were chilled at 30° F., instead of at 25° F. This second

procedure appeared to be more satisfactory than the first and duplicate tests on stability of the processed materials checked more closely.

The effect of the presence of water on the activity of antioxidants representative of those permitted for use in lard by the Canadian Food and Drugs Act (1947) was investigated. (The stabilizers listed in Table III were tested in all six experimental lards, which were adjusted to zero and 1.5 or 1.6% moisture. Some of the antioxidant concentrations (0.5%) were higher than would be used in actual practice: the Act permits the addition of only 0.2% of stabilizing materials, either singly or in combination; moreover, 0.5% gum guaiac or lecithin gives a marked increase in lard color and affects flavor adversely. However, it was considered desirable for comparative purposes to use the larger quantities in initial tests. Citric acid and alpha-tocopherol were used at several levels; low optimum concentrations for these antioxidants have been noted (13). The water content of the wet lard was chosen to be definitely higher than that encountered in commercial lard (less than 1%), but not so high as to facilitate the growth of micro-organisms. Antioxidants were added prior to blending.

All samples were stored at 90° F., to approximate severe shelf storage conditions, and to permit fairly rapid deterioration of the lard while maintaining it in an emulsified state. At each sampling the contents of each jar was stirred thoroughly with a stout glass rod and approximately 10 gm. removed and placed in a sealed container, which was held at −40° F. until chemical measurements could be made.

Measurements

Determination of moisture by the vacuum oven method (1) was impractical at moisture contents higher than 1% because of spattering and the lengthy heating required. The higher moisture values were therefore calculated from measurements of the saponification numbers of the lard samples (1).

There was some doubt as to the suitability of different assessments of rancidity, since the effect of high water levels on the chemical tests was not known. Peroxide oxygen and alpha-dicarbonyl values were determined, since these were known to be useful estimates of oxidative spoilage and to be highly correlated with organoleptic ratings (7). Free fatty acid content was also measured, since added water above 3% might increase hydrolytic spoilage by micro-organisms (5). The following changes in the usual methods (7) were made: for the peroxide oxygen procedure, an accurately weighed 1 gm. (± 0.05 gm.) sample was used; and for the alpha-dicarbonyl measurement, the test solution was filtered through a Whatman No. 40 paper before the colorimetric reading was taken.

Samplings were made at two- or three-week intervals. Storage life was estimated as the time in weeks at 90° F. for the lard to attain a peroxide oxygen value of 10 ml. of 0.002 *N* sodium thiosulphate per gm. (7). The statistical significance of these keeping times was assessed by analyses of variance.

Results

Effect of Moisture Content

In a preliminary experiment, single samples of the four selected lards (*A*, *B*, *C*, and *D*) prepared by Blending Procedure I to contain 0, 1.5, 3, 6, and 12% moisture were stored at 90° F. Contrary to expectations, there was no marked increase in free fatty acid content of any of the samples. A count

TABLE I

EFFECT OF DIFFERENT MOISTURE LEVELS ON THE STORAGE LIFE OF SELECTED LARDS AT 90° F.

(Blending Procedure I, under nitrogen)

Water content, %	Storage life, weeks at 90° F.				
	Wet rendered		Dry rendered		Average
	Lard *A*	Lard *B*	Lard *C*	Lard *D*	
Untreated (0.1)	1.1	5.4	3.2	3.9	3.4
0.0	0.7	5.4	3.3	3.8	3.3
0.75	1.9	4.2	4.0	2.3	3.1
1.5	2.5	5.2	2.1	3.8	3.4
3.0	2.3	5.4	3.0	4.0	3.7
6.0	1.3	3.3	2.0	2.2	2.2
12.0	0.2	2.8	2.6	2.6	2.0
Average	1.4	4.5	2.9	3.2	

Necessary differences (5% level of statistical significance):
between averages for different lards, 0.8 week;
between averages for different moisture levels, 1.0 week.

TABLE II

EFFECT OF DIFFERENT MOISTURE LEVELS ON THE STORAGE LIFE OF COMPOSITE LARDS AT 90° F.

(Blending Procedure II, under air)

Water content, %	Storage life, weeks at 90° F.		
	Lard *E*	Lard *F*	Average
0.0	4.5	3.3	3.9
0.2	4.9	2.5	3.7
0.4	4.2	2.9	3.6
0.8	3.8	3.3	3.6
1.6	3.0	3.2	3.1
3.2	4.8	2.9	3.9
6.4	4.5	3.8	4.2
12.8	2.6	1.8	2.2
Average	4.0	3.0	

Necessary differences (5% level of statistical significance):
between averages for different lards, 0.6 week;
between averages for different moisture levels, 1.2 weeks.

for micro-organisms showed less than 10 per ml. in representative samples after 10 weeks' storage. Peroxide oxygen and alpha-dicarbonyl values increased in a fairly regular manner to give induction-type curves, and values for these two tests were closely correlated ($r = .96$), in agreement with previous findings (7). This trial indicated that spoilage might be followed by determination of peroxide values only, that low moisture levels should be more carefully examined, and that duplicate samples should be prepared for each moisture level.

In a more comprehensive experiment with the same materials, using the same blending technique (I), moisture contents of 0, 0.75, 1.5, 3, 6, and 12% were studied. The storage results indicated a significant reduction in keeping quality at moisture values of 6 and 12% (Table I). All the lards appeared to react to incorporated water in a similar manner, although they differed in initial stability.

TABLE III

EFFECT OF DIFFERENT ANTIOXIDANTS ON THE STORAGE LIFE OF SELECTED LARDS AT 90° F.

(Blending Procedure I, under nitrogen)

Sample treatment	Storage life, weeks at 90° F.				
	Wet rendered		Dry rendered		
	Lard *A*	Lard *B*	Lard *C*	Lard *D*	Average
Antioxidants in dry lard (zero moisture):					
None*	0.7	5.4	3.3	3.8	
0.005% citric acid	0.9	7.3	2.0	5.4	
0.05% citric acid	2.3	9.7	2.3	4.3	
0.5% citric acid	1.8	12.9	2.8	12.6	9.0
0.5% gum guaiac*	44.0	44+	44+	44+	
0.5% lecithin*	11.2	31.0	24.3	28.4	
0.05% alpha-tocopherol	12.7	18.4	13.5	18.0	
0.5% alpha-tocopherol	16.3	9.9	13.3	13.3	
Antioxidants in wet lard (1.5% moisture):					
None*	2.5	5.2	2.1	3.8	
0.005% citric acid	1.8	6.9	3.4	3.4	
0.05% citric acid	1.3	6.5	3.3	4.3	
0.5% citric acid	2.0	6.6	3.8	3.8	6.4
0.5% gum guaiac*	44+	44+	44+	44+	
0.5% lecithin**	—	—	—	—	
0.05% alpha-tocopherol	7.2	12.9	10.4	12.9	
0.5% alpha-tocopherol	9.1	9.3	8.2	10.8	
Average	5.5	10.0	6.3	8.9	

Necessary differences (5% level of statistical significance):
between averages for wet and dry lards, 1.0 week;
between averages for different lards, 1.4 weeks;
between any two samples, 4.6 weeks.

*Not included in calculations.

**Contained visible mold growth at later storage periods, hence true storage life could not be estimated.

With the two composite lards (*E* and *F*), using the revised blending procedure (II), the effect of added water was tested at 0, 0.2, 0.8, 1.6, 3.2, 6.4, and 12.6%. The results, shown in Table II, were similar to those of the preceding experiment, except that only the 12% moisture level caused a significant reduction of storage life.

Effect of Antioxidants

Keeping times for selected lards containing antioxidants are shown in Table III. Samples containing 0.5% gum guaiac were very stable and peroxide

TABLE IV

EFFECT OF DIFFERENT ANTIOXIDANTS ON THE STORAGE LIFE OF COMPOSITE LARDS AT 90° F.

(Blending Procedure II, under air)

Sample treatment	Storage life, weeks at 90° F.			
	Lard *E*	Lard *F*	Average	Grand average
Antioxidants in dry lard (zero moisture):				
None*	4.5	3.3	3.9	
0.02% citric acid	6.5	6.4	6.5	
0.2% citric acid	11.9	8.2	10.1	
0.02% gum guaiac	29.4	17.9	23.7	
0.2% gum guaiac*	39+	33.9	—	12.7
0.02% lecithin	8.8	5.4	7.1	
0.2% lecithin	13.7	10.6	12.2	
0.02% alpha-tocopherol	18.9	14.7	16.8	
0.2% alpha-tocopherol	16.2	9.8	13.0	
Antioxidants in wet lard (1.6% moisture):				
None*	3.0	3.2	3.1	
0.02% citric acid	6.4	3.6	5.0	
0.2% citric acid	6.1	3.4	4.8	
0.02% gum guaiac	30.1	17.2	23.7	
0.2% gum guaiac*	39+	39+	—	8.7
0.02% lecithin	5.9	3.3	4.6	
0.2% lecithin	8.2	4.3	6.3	
0.02% alpha-tocopherol	11.7	9.1	10.4	
0.2% alpha-tocopherol	9.0	5.7	7.4	
Average	13.0	8.6		

Necessary differences (5% level of statistical significance):
between averages for different lards, 1.1 weeks;
between grand averages for wet and dry lards, 1.1 weeks;
between averages for sample treatments, 2.0 weeks.
**Not included in calculations.*

determinations were not continued to the limiting value. Wet lard (1.5% moisture) with 0.5% lecithin gave erratic results and contained visible molds at the later samplings. Hence some of the values could not be estimated.

With incorporated antioxidants, all four lards kept better at zero moisture than at 1.5% moisture (Table III). Over all antioxidant treatments, the

average storage life of lards *B* and *D* was longer than that of *A* and *C*. On the whole, alpha-tocopherol gave longer life than citric acid, particularly in dry lard; the difference in effectiveness was greatest in the most unstable lard (lard *A*) and least in the most stable lard (lard *B*). Better results were obtained from 0.05% than from 0.5% of alpha-tocopherol in the better lards (*B* and *D*) but not in the poorer lards (*A* and *C*). No significant difference in the results from the three levels of citric acid was demonstrable, although there was some indication that citric acid was more effective at the higher concentrations in the better (dry) lards. Gum guaiac was the most effective antioxidant at the 0.5% level, with lecithin next (for dry lard). No consistent difference in the results associated with wet or dry *rendering* of the lards was detectable.

In further experiments to test the relative effectiveness of the above stabilizers in composite lards, the results (Table IV) were confirmatory of the earlier experiments. Analysis of variance of the data indicated that, on the average, the two lards differed significantly in storage life, and that the dehydrated samples of both lards kept better than the ones with added moisture. The experiment did not demonstrate any significant difference between the two lards in their response to the antioxidants. The storage life of both lards was definitely prolonged by gum guaiac and alpha-tocopherol, both of which gave a longer storage life than citric acid or lecithin. A somewhat longer storage life resulted from the higher of the two concentrations of citric acid and lecithin tested, whereas with alpha-tocopherol the lower concentration gave the longer storage life.

Conclusions and Discussion

The results emphasize the importance of the nature of the substrate and the concentration of the addend in lard stabilization. Water alone reduced the storage life of lard only at the higher moisture levels (6 and 12%), but the added stabilizers were more effective in dry lard than in lard containing about 1.5% added water, with the exception of gum guaiac, which behaved similarly in the presence or absence of water. At the 0.5% level, lecithin was more effective than alpha-tocopherol, but at the lower concentrations the reverse was true. Alpha-tocopherol showed a low optimum concentration effect, which tended to be obscured with the lards of poorer initial quality. Citric acid, on the other hand, appeared to be more effective at the higher levels of incorporation in the better (dry) lards, but exhibited no concentration effects in the presence of moisture. The stabilization imparted by gum guaiac and lecithin increased with the amount added. Gum guaiac appeared to be the most generally effective antioxidant permitted for use in Canadian lard by the Food and Drugs Act (1947).

The results for the effect of water alone at small concentrations, and for the relative activities of citric acid and gum guaiac in the presence and absence of water are in agreement with earlier findings (6, 8, 10). Optimum concen-

trations for citric acid, lecithin, and alpha-tocopherol have also received earlier study (2, 13, 17). The action of water on the stabilizing power of lecithin and alpha-tocopherol has apparently not been specifically examined heretofore.

Since gum guaiac and alpha-tocopherol are both phenolic in nature, it might be expected that the antioxidant activity of both materials would be reduced in the presence of water (12, p. 174). The undiminished effect of gum guaiac in lard containing water may be ascribed to the complex nature of the guaiac resin: reduction in potency of the contained phenols by water may be offset by hydrolysis of inactive ester or oxy groups to more active structures (17).

As the stabilizing action of citric acid was reduced by water, this action apparently does not depend on ionization. If citric acid is considered as a synergist only, dependent for its activity on traces of naturally occurring phenolic antioxidants (17), its lessened activity in the presence of water may be due in turn to the lessened activity of these phenolic compounds in the presence of water. This is supported by the report that the chief naturally occurring antioxidants in lard are tocopherols (4; 16, p. 18).

The most satisfactory method of preparing a stable, dehydrated butter oil was found to be one that ensured the transfer of a high phosphatide content from the serum to the fat (5). Consequently, the reduction in antioxidant activity of lecithin in the presence of water may be due to a reversal of this effect; i.e., transfer of phosphatide to the aqueous phase.

The most evident demonstration of an optimum antioxidant concentration occurred with alpha-tocopherol. With both lecithin and alpha-tocopherol excess amounts are detrimental because these materials are vulnerable to oxidation, and their oxidation products accelerate spoilage of the fat (16, p. 19; 17). The optimum concentration of alpha-tocopherol was higher with the poorer lards presumably because these initially contained more pro-oxidant materials that destroyed more of the added antioxidant. The optimum concentration of citric acid may be related to its synergistic action (17); addition of further citric acid after maximum synergistic effect was obtained would be useless.

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A COMPARATIVE STUDY OF TEN ELECTRICAL METERS FOR DETERMINING MOISTURE CONTENT OF WHEAT[1]

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Abstract

Ten electrical moisture meters were tested with 159 samples of Canadian hard red spring wheat, of Grades 1, 2, 3, and 4 Northern, representing a moisture range of 11 to 17%. The two-stage vacuum oven and Brown–Duvel methods were used as bases of comparison. Regression equations, standard errors of estimate, and data on temperature effects are reported. The standard error of estimate of vacuum oven results was 0.15% for the Brown–Duvel method, 0.23% for the Tag–Heppenstall meter, and 0.28% for the Universal meter. Other meters had higher errors of estimate.

In 1934, four electrical meters for determining the moisture content of grain were studied in this laboratory (2, 3, 4). Of these, the motor-operated Tag–Heppenstall meter was found to be the most accurate and has since been used in the Inspection Branch of the Board of Grain Commissioners for preliminary tests of moisture content. The results are accepted for samples having moisture contents 0.3% below the maximum limit for straight grades. In normal years about 90% of all samples enter this category. The remaining samples are retested by the Brown–Duvel oil-distillation method and are graded straight, tough, or damp, in accordance with the results of this test.

Since 1934, and particularly within the last two or three years, a number of new meters have been placed on the market. The possibility that one or more of these might prove useful at some stage of the grain marketing process in Canada merited investigation. Accordingly, through the courtesy of the manufacturers, which is gratefully acknowledged, models of the meters were borrowed for the study reported in this paper. Names, manufacturers, and countries of origin of the meters, are listed below:

Name	*Manufacturer*	*Country*
Universal Moisture Tester Model 4A No. 59	R. H. Nichols Ltd., Toronto	Canada
G.R.L. Moisture Meter	Grain Research Laboratory, Winnipeg.	Canada
Tag–Heppenstall Moisture Meter S-3604	C. J. Tagliabue Corporation, (N.J.), Newark, N.J.	United States
Tag–Dielectric Moisture Meter S-Cm-202	C. J. Tagliabue Corporation (N.J.), Newark, N.J.	United States

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[2] *Chemist.*
[3] *Senior Technician.*
[4] *Chief Chemist.*

Patterson Moisture Meter Type 100A S-4	C. J. Patterson Company, Kansas City, Mo.	United States
Steinlite Moisture Meter S-9894	Fred Stein Laboratories, Atchison, Kansas.	United States
Mullard Moisture Meter Type E910 No. RT-279	Mullard Electronic Products Ltd., Shaftesbury Ave., London.	England
N.P.L. Moisture Meter No. 28145	Baldwin Instrument Co. Ltd., Dartford, Kent.	England
Marconi Moisture Meter Type TF842 No. 705713	Marconi Instruments Ltd., St. Albans, Herts.	England
Toplis–Simpson Moisture Meter S-1300/08	Toplis, Simpson & Co. Ltd., Wembley, Middlesex.	England

Perhaps it should be mentioned at once that some of the meters are still under development, so that parts of this paper may be out of date before it is published. For instance: the Universal meter that was studied had a screw press, but a model with a hydraulic press has since been produced and may finally be preferred; the G.R.L. meter, an experimental model, is not yet being manufactured; the Patterson meter, which is primarily designed for flour, is now used with ground rather than whole wheat; and improved models of the Toplis–Simpson and Marconi meters have been developed since the present investigation was completed. Moreover, news has recently been received of other meters that may shortly be on the market. However, since further investigations are not planned for the immediate future, there is no point in delaying publication of this paper.

Two main investigations are described. The first involved calibration, with 159 samples of wheat at 72° F., of all meters and the Brown–Duvel method in terms of the vacuum oven method. The second dealt with correction factors for temperature, and was made with four samples of different moisture contents at five temperature levels. Materials and methods for both studies are described in the next section. Data for the comparison of the Brown–Duvel and vacuum oven methods are then given. Following sections deal with the operation of the meters and then with the individual meters in order of their estimated precision. A final section contains a general discussion of the results.

Materials and Methods

Materials

One hundred and fifty-nine samples of Western Canadian hard red spring wheat, each taken from an individual carlot shipped during the 1947-48 crop year, were obtained through the co-operation of the Inspection Branch of the Board of Grain Commissioners. The series was selected so as to obtain a relatively uniform distribution of moisture contents between 11.5 and 17.5%

and of bushel weights between 55 and 65 lb., and was restricted to Grades 1, 2, 3, and 4 Manitoba Northern. Individual samples weighed about 7.5 kgm. and were received and kept in 1-gal. tin cans with small screw stoppers.

Calibration Study

All meter tests were made by one man with an assistant to help with the subdividing and weighing of the samples. Moreover, this team made a preliminary study with 50 samples in order to familiarize itself with the operation of the meters. The work was done in a small, 14 by 11 ft., laboratory automatically maintained at 72° ± 0.5° F. As the air in this laboratory circulates rapidly, and as the control cycle is approximately five minutes, such minor variations as occur in temperature are too rapid to be effectively transferred to equipment or samples. Throughout the investigation, voltage of a-c. current may have varied between 114 and 118 v., but tests showed that none of the meters were affected by variations of as much as ± 10 v.

All meters were tested with one of the 159 main samples before starting tests with another. The 7.5 kgm. sample, which had been kept in the constant temperature laboratory for at least two days, was first mixed by passing it four times through a Boerner sampler and was subsequently divided into 64 subsamples of 115 gm. each. Two subsamples were then allotted at random to each meter, to the Brown–Duvel method, and to the vacuum oven method. When a meter required more than 115 gm., additional pairs of subsamples were allotted to it and composited to make larger subsamples. Single readings on duplicate subsamples were made with each meter, and the order of testing the meters was randomized. About 45 min. was required to prepare subsamples and to take all meter readings on one main sample.

Temperature Study

Four large samples (35.5 kgm.) were obtained with the following average moisture contents: 16.0, 14.8, 13.6, and 11.5%. Each of these was thoroughly mixed in a McClellan mixer and then subdivided with the Boerner sampler into five 7.5-kgm. samples, one of which was used at each of five temperatures, 50°, 60°, 70°, 80°, and 90° F. The procedure described above was used for obtaining subsamples and making the meter readings.

The temperature of the laboratory was set at 50° F. for the first working period and was subsequently raised 10° F. for each of four additional working periods. After the temperature had been raised by 10° F., the next working period was delayed about 40 hr. to ensure that meters and samples reached equilibrium at the new temperature. Tests showed that no significant loss of moisture from the samples occurred during the 11 days required for this study.

Vacuum Oven and Brown–Duvel Methods

A two-stage vacuum oven method (2) was employed as a primary standard procedure, and determinations were started on the day that meter readings were made on the sample. The duplicate subsamples allotted to the vacuum

oven were air-dried at room temperature in trays for two days during which the moisture content was reduced to about 9%. After grinding in a Wiley mill with a 1 mm. sieve, duplicate 2-gm. samples of the meal were subsequently dried overnight in the vacuum oven at 100° C. Four results were thus obtained for each of the 159 main samples. The standard error of the mean of these four determinations was 0.047%.

The Brown–Duvel tests were made by an experienced operator using a carefully standardized, six compartment, electrically heated machine, with a 100 gm. sample and a cutoff temperature of 185° C. The standard error of the mean of tests on duplicate subsamples was 0.05%. It was the intention

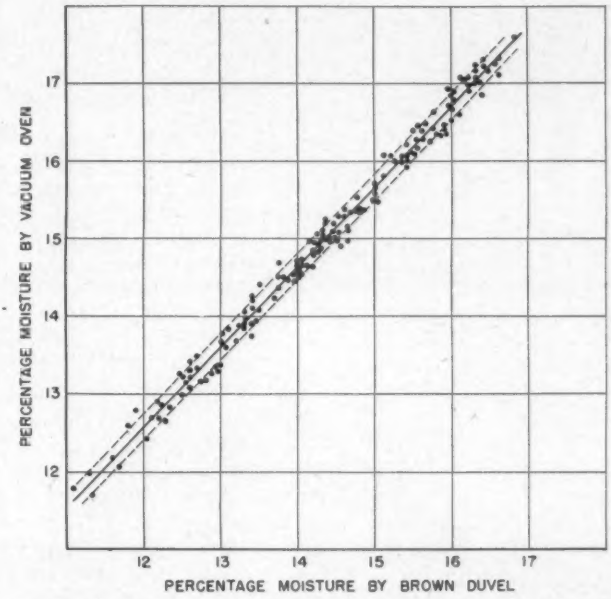

FIG. 1. *Scatter diagram showing the relation between moisture contents determined by the vacuum oven and Brown–Duvel methods for 159 samples of hard red spring wheat.*

to make Brown–Duvel determinations on the same day as meter readings, but this did not prove feasible. Accordingly, some samples were held in glass jars with screw caps for several weeks; but a check of sample weights showed that no appreciable change in moisture had occurred during this period.

Fig. 1 is a scatter diagram in which oven results are plotted against Brown–Duvel results for the 159 samples of hard red spring wheat. This diagram shows that the 159 samples covered the range of moisture from 11.5 to 17.5% relatively uniformly. Since the dots fall relatively close to the regression line, the agreement between the two methods is shown to be good. The error of prediction of vacuum oven moistures from Brown–Duvel results

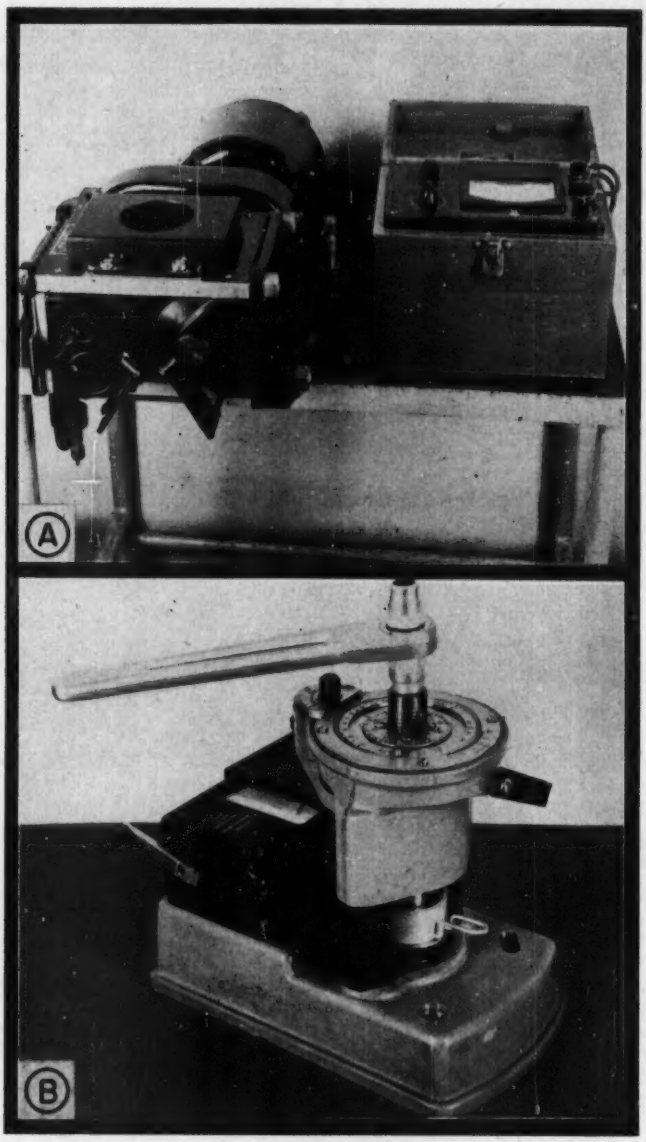

FIG. 2. *Photographs of (A) Tag–Heppenstall Meter and (B) Universal Meter.*

proved to be 0.15% moisture, and dotted lines corresponding to this deviation about the regression line are also shown in the figure. It may be mentioned, in passing, that the error of estimate for the Brown–Duvel method proved to be considerably lower than that for any of the electrical meters.

Two further points should be noted. Firstly, the regression line in Fig. 1 does not make an angle of 45° with the axes; the regression coefficient is 1.03 rather than 1.0. This means that, by comparison with vacuum oven, the Brown–Duvel method underestimates moisture content by an amount that increases with increasing moisture level. And secondly, the average moisture for the 159 samples was 15.0% by the vacuum oven, whereas it was 14.4% by the Brown–Duvel method. This discrepancy arises from the fact that the Brown–Duvel method was originally calibrated against an air-oven method. Cook, Hopkins, and Geddes (2) obtained similar results. In spite of these faults, the Brown–Duvel method gives precise results that can readily be converted to vacuum oven moisture by means of the regression equation $y = 1.03x + 0.14$ where y is the vacuum oven moisture and x is the Brown–Duvel moisture. It should be added that no correction for the temperature of the grain is required with the Brown–Duvel method, whereas all electric meters have relatively large temperature coefficients.

Operation of the Meters

Photographs of all the meters except the Marconi and the Toplis–Simpson are shown in Figs. 2 and 3. No attempt will be made to describe the circuits in detail, but notes on the principal differences in design and operating technique are given in this section.

The Tag–Heppenstall and Universal meters measure the conductance or resistance of the grain and can be calibrated in ohms. All other meters make measurements that are functions of the change in capacitance of a condenser when grain is introduced as a dielectric. In three of the meters, the Universal, G.R.L., and Patterson, the characteristics of the circuits are such that no standardization of them is required. The remaining meters have convenient provisions for standardizing or balancing the circuits, and this operation must be undertaken before a series of tests is made. All meters require a-c. current of either 110 or 220 v., except the Toplis–Simpson and Marconi meters, which have batteries, and the Universal meter, which requires neither power nor batteries. The Universal meter has a built-in armored thermometer, but with all other meters the temperature of the grain must be read with a thermometer before starting the test.

Resistance Type Meters

In the Tag–Heppenstall meter (Fig. 2, A), two corrugated steel rolls serve as electrodes; one is motor-driven and the other idles. Grain is poured into the hopper above the rolls and is partially crushed as it passes between them. A selector switch is turned to one of eight positions that control the range

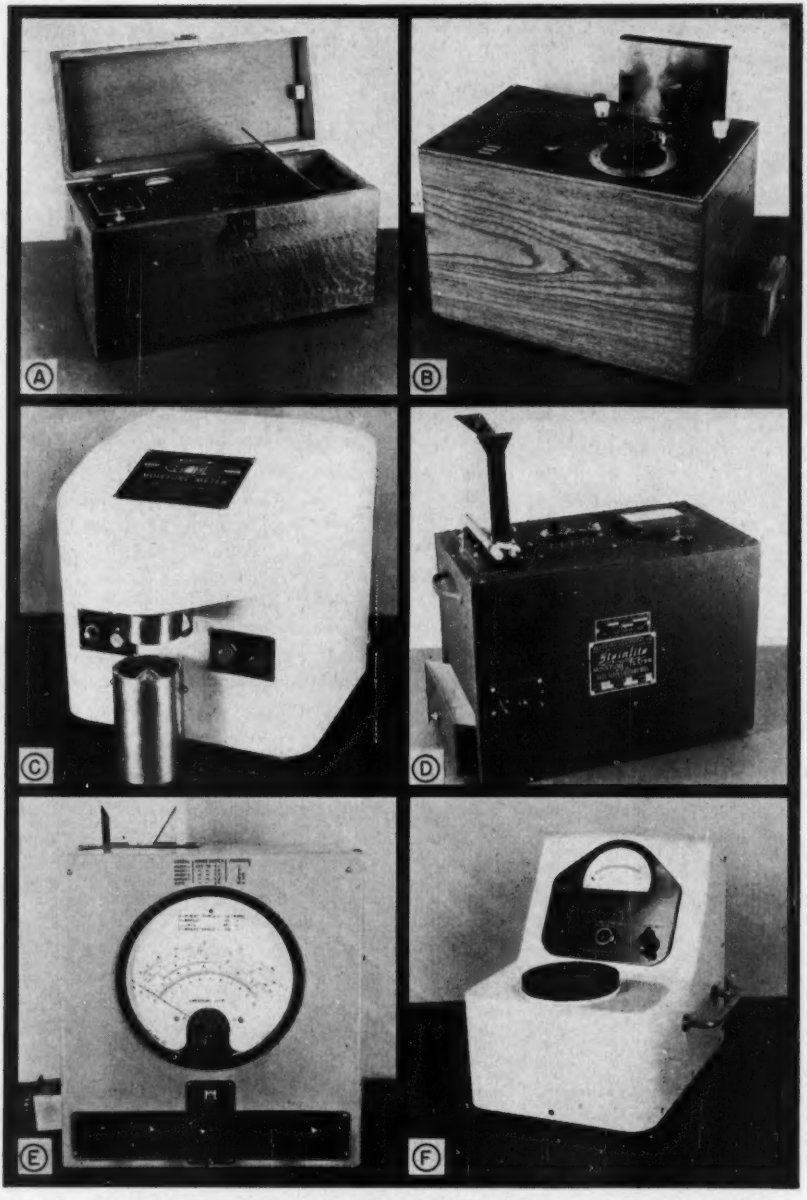

FIG. 3. *Photographs of (A) Tag–Dielectric, (B) G.R.L., (C) Patterson, (D) Steinlite, (E) Mullard, and (F) N.P.L., moisture meters.*

of values indicated by the galvanometer, and the position of the galvanometer pointer is read. Charts are provided for converting readings to moisture contents. Only about 10 to 20 sec. is required to make a reading.

The spacing between the rolls is critical and must be maintained with shims when the rolls wear appreciably as a result of passing several thousand samples through them. An increase of 1/1000 in. in the spacing reduces moisture readings by about 0.03%. The same spacing is used for wheat, barley, and oats, but a thick shim must be introduced to narrow the spacing for testing flax. A different set of rolls with larger corrugations is used for corn.

In the Universal meter (Fig. 2, B), a hand-driven megger establishes a voltage across a sample of compressed grain, and electrical resistance is indicated on an ohmmeter of the dynamometer type. The instrument thus requires neither batteries nor power supply.

A test is made on a 10 gm. sample weighed to the nearest kernel. This is transferred to a steel cup with a cylindrical plastic lining (internal diam. 2 in.). The cup is placed beneath the ram of a screw press operated with a ratchet wrench, and the grain is compressed to a thickness of 0.220 in., which is indicated by a micrometer device on the ram. The megger is then cranked rapidly and the meter and temperature are read. After the test, which requires about 1.5 min., the partially crushed grain can be readily removed from the cup with a screw driver.

Circular sliding scales for converting temperatures and meter readings to moisture content are provided on the top of the press. But the calibration does not appear to be correct for wheat (see next section) and would not be expected to suit all types of grain and seeds. Accordingly, conversion charts will probably be required. The meter can be used with a variety of grain and seeds, such as flax and rape, without change in adjustment. All that is required is that the weight of sample and the thickness of the pressed layer be adjusted to suitable values for each seed.

Dielectric Type Meters

All the dielectric meters are operated in much the same way in that a sample is introduced into the cell, certain switches, push buttons, or dials are manipulated, and a reading is taken either from a dial or from an indicating instrument with pointer and scale. Single tests can be made in about 1 to 1.5 min. Photographs of six of the meters are shown in Fig. 3: (A) Tag–Dielectric; (B) G.R.L.; (C) Patterson; (D) Steinlite; (E) Mullard; and (F) N.P.L. The principal differences between these meters are noted below. Most of the meters have already been described: G.R.L., (6); Marconi (1); N.P.L., (5); and the remainder in advertising literature.

In order to obtain uniform packing of the sample within the test cell, loading hoppers with spring or trip release mechanisms are provided in the G.R.L., Steinlite, Mullard, and Marconi meters. Each of these has provision for

dumping the tested sample into a drawer for removal. A special funnel with a spring-operated release is provided for loading the cell of the Patterson meter. And this and the remaining meters have removable cells that are readily emptied.

No loading hoppers are provided for the Tag–Dielectric, N.P.L., and Toplis–Simpson meters. Tests showed that uniform loading of the cells of the last two meters did not improve the results obtained with them. But uniform loading did improve the results obtained with the Tag–Dielectric meter, and this meter was therefore used with the Steinlite hopper.

The weights of wheat required for single tests with the dielectric meters are as follows: Tag–Dielectric, 260 gm.; G.R.L., 190 gm.; Patterson, 20 gm.; Steinlite, 150 gm.; Mullard, 100 gm.; N.P.L., about 300 gm.; Marconi, about 150 gm.; and Toplis–Simpson, about 150 gm. With the last three meters, the cell or hopper is merely filled level with the top. With all other dielectric meters, the sample must be weighed.

The Patterson meter has a novel feature consisting of a servo-mechanism that balances the circuit. After the loaded cell is clipped in place, a button is pushed, and the operator has only to read the position of a scale when the servo-mechanism stops.

Results

Sampling error, in terms of the electrical properties measured, proved to be negligible; each meter gave almost identical results for duplicate subsamples. Accordingly, the statistics reported below relate to the mean values given by the meters and by the vacuum oven for each of the 159 main samples. Scatter diagrams, regression equations, standard errors of estimate of moisture content (vacuum oven) from meter readings, and graphs showing the results of the temperature study, are presented for the more precise meters, and corresponding data are summarized for the remaining ones.

Tag–Heppenstall Meter

The Tag–Heppenstall meter can be most conveniently calibrated in ohms. It has a range from 2000 ohms to about 85 megohms, which corresponds to a moisture content of wheat ranging from 10 to 24%. An inverse logarithmetic relation exists between the resistance (R) of the grain in ohms and percentage moisture content. Accordingly, a direct linear relation is obtained by plotting moisture content against $10-\log R$. Fig. 4 shows this plot for the 159 samples tested. The slope of the regression line is 2.47 and the standard error of estimation of moisture content (vacuum oven) from the meter readings is 0.23% moisture. The dotted lines drawn 0.23 percentage units above and below the regression line thus represent the error of estimate and include about two-thirds of the points. Points lying farther from the regression line represent samples whose moisture contents would be least accurately estimated by the meter on the basis of the regression equation. Errors of as much as $\pm$ 0.7% can occasionally occur.

The regression equation obtained for the Tag–Heppenstall meter takes the form $y = bx + c$, where y is the moisture content, x is the meter reading in log ohms (or 10–log ohms), b is the regression coefficient, and c is a constant. The value for c is a function of the sharpness of the corrugation on the rolls and the space between the rolls. Accordingly, exactly the same equation is not obtained for two meters unless the rolls and spacing are identical. For

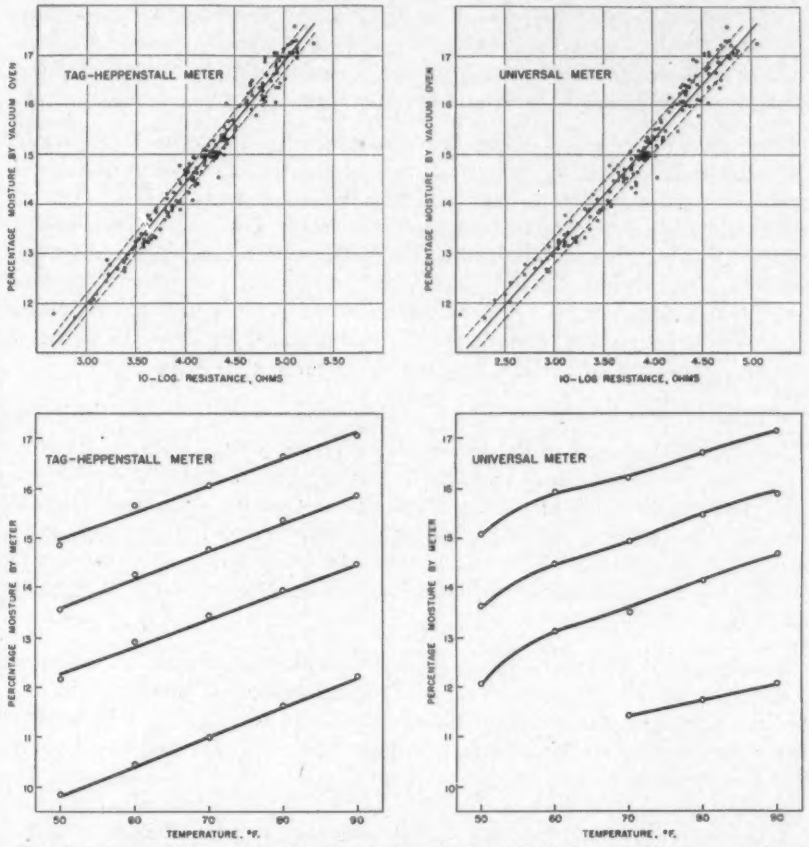

FIG. 4. *Scatter diagrams showing the relations between moisture content (vacuum oven) and 10−log resistance (ohms) for Tag–Heppenstall and Universal meters. Curves showing the relations between percentage moisture by meter and temperature for the two meters.*

this reason, a direct comparison of the equation obtained in the present study and that obtained in a similar study (3), made in the laboratory 15 years ago, is not feasible. However, indirect comparison shows that if both meters had been adjusted to give identical readings for grain of 15% moisture, results obtained with the two meters at 11 or 17% moisture would have differed by 0.03%. This difference is negligible for all practical purposes.

As the rolls on the meter wear and the spacing between them increases, moisture content is progressively underestimated. This error can be corrected by shimming when a decrease in spacing of 1/1000 in. raises the meter reading by the equivalent of about 0.03% moisture. However, the most accurate corrections for wear cannot be made merely by re-establishing a constant spacing between rolls. For experiments have shown that the spacing required to maintain the calibration also depends on the sharpness of the corrugations; as this decreases, compensation must be afforded by additional decrease in spacing. These minor difficulties are readily overcome in practice by calibrating meters periodically against the vacuum oven, or other standard method, and shimming to re-establish the calibration.

The results for the study of the effect of temperature on the Tag–Heppenstall meter are shown in Fig. 4. The four curves, representing samples of different moisture contents, are linear and parallel. This represents an ideal situation in which a single correction factor covers the temperature range (50° to 90° F.) and moisture range (11.5 to 16.0%) investigated. It seems probable that this generalization will hold for wider ranges. The regression of moisture on temperature is 0.056% per ° F. This is lower than the value of 0.068% established in the earlier study (3), and since this was obtained by study of about 30 samples, rather than four, it is considered the more reliable figure.

Universal Meter

Although the megger of the Universal meter is provided with an arbitrary scale, this can readily be converted to ohms. The range of the megger is from 5,000 ohms to 50 megohms, which corresponds to a moisture range of about 11.5 to 18%. The scatter diagram for moisture content (vacuum oven) against $10-\log R$ (Fig. 4) shows an essentially linear relation represented by the regression equation, $y = 2.24x + 6.37$. The standard error of estimate is 0.28%.

Since the Universal meter shows considerable promise, it was also calibrated against the Brown–Duvel method. The linear regression proved to be $y = 2.15x + 6.06$, and the standard error of estimate was 0.27%. To make a direct comparison of the arbitrary megger scale readings and the Brown–Duvel method, a fourth degree polynomial was fitted to the points. The reduction in the standard error by this method of calculation was negligible. A chart for converting readings on the arbitrary megger scale to moisture contents, based on the fourth degree equation, has been prepared.

Results for the study of temperature effects are shown in Fig. 4. The curves suggest that a single correction factor (0.045% per ° F.) is applicable within the range of 60° F. to 90° F. and of 15.7% down to 13.0%, and possibly to 11.0%, of moisture. There is an indication, however, that the slopes of the curves change below 60° F., and that different corrections would be required below this temperature level. Further investigation is required to establish this point.

The manufacturers have calibrated the instrument, and have provided engraved scales on the top of the instrument from which the operator can readily obtain the moisture content when the megger reading and temperature are known. This calibration does not agree with those obtained in the present study either for the meter and the vacuum oven or for the meter and the Brown–Duvel method. By comparison with our vacuum oven data, the manufacturers' calibration is lower by an average of 0.75%; by comparison with our Brown–Duvel data, it is high at low moisture levels (11 to 13%) and low at high moisture levels (14 to 17%). In view of the large number of samples used in the present study, and the fact that all these were taken from commercial carlots of grain, it seems probable that the manufacturers' calibration is erroneous.

Dielectric Type Meters

Scatter diagrams for six of the dielectric type meters are shown in Fig. 5. The calibration equations and standard errors of estimate of moisture content (vacuum oven) are given in Table I. Diagrams for the Tag–Dielectric and

TABLE I

CALIBRATION EQUATIONS AND STANDARD ERRORS OF ESTIMATE FOR ELECTRICAL MOISTURE METERS

Meter	Unit of measurement	Calibration equation*	Standard error of estimate, moisture, %
Tag–Heppenstall	10 − log of resistance, ohms.	$y = 2.47x + 4.54$	0.23
Universal	10 − log of resistance, ohms.	$y = 2.24x + 6.37$	0.28
Tag–Dielectric	Meter reading	$y = .109x + 3.62$	0.35
G.R.L.	Meter reading	$y = .132x + 10.07$	0.36
Patterson	Meter reading	$y = .186x + 7.29$	0.41
Steinlite	Meter reading	$y = .106x + 11.09$	0.44
Mullard	Meter reading	$y = .876x + 10.88$	0.48
N.P.L. Switch Pos. 1	Meter reading	$y = .116x + 14.57$	0.50
N.P.L. Switch Pos. 3	Meter reading	$y = .131x + 9.25$	0.49

*y = *Vacuum oven moisture.* x = *Unit of measurement.*

Patterson meters suggest slightly curvilinear relations especially for moisture contents over about 16%. The N.P.L. meter is provided with a selector switch for changing the range, and diagrams for two ranges are therefore shown.

Results obtained with the Marconi (Model TF842) and the Toplis–Simpson (Model S–1300/08) meters were less satisfactory than those for the other meters. As both companies have substituted new models for the older ones examined in this laboratory, data on the latter are not reported.

Fig. 6 shows the results of the temperature study for six meters. Among these, the G.R.L. meter most nearly approaches the ideal in which all curves are straight and parallel. But the Tag–Dielectric, Steinlite, and Mullard

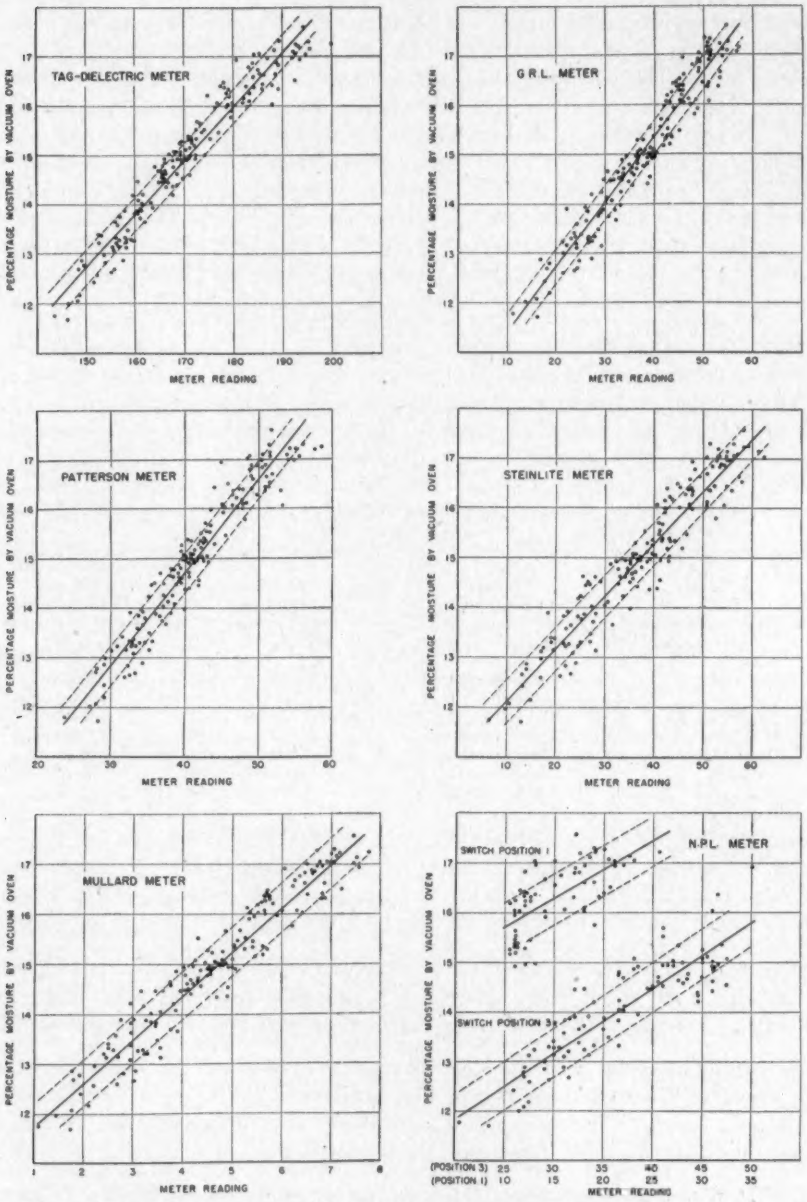

FIG. 5. *Scatter diagrams showing the relations between percentage moisture (vacuum oven) and meter readings for six electrical moisture meters.*

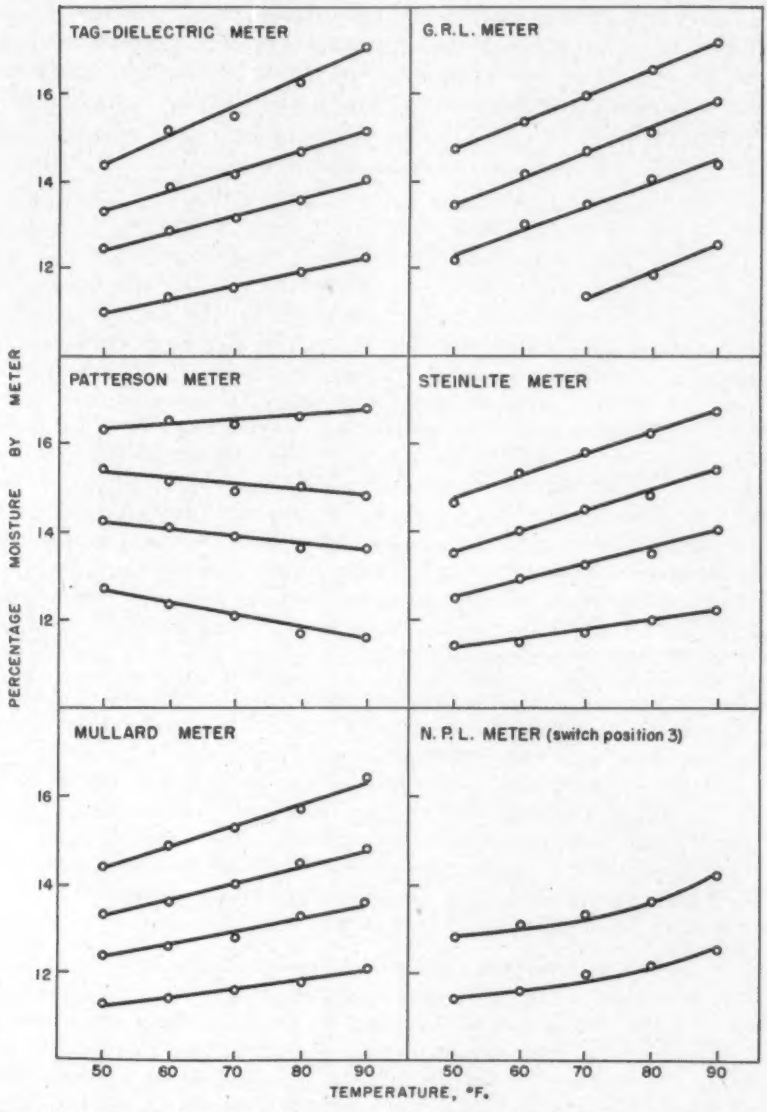

FIG. 6. *Curves showing the relation between percentage moisture by meter and temperature for six electrical moisture meters.*

meters show only minor deviations from the ideal. The N.P.L. meter shows slightly curvilinear relations. On the other hand, the Patterson meter appears to require different correction factors for samples of high and low moisture content. In general, however, the adoption of a single temperature correction factor for each meter would introduce errors that are small by comparison with those resulting from the looseness of the relation between moisture content and meter readings.

Discussion

The data reported in this paper relate to single samples of each manufacturer's meter. A question thus arises as to whether a generalized statement can be made about the comparative merits of the meters. For the Tag–Heppenstall and Universal meters, several of which have been tested in this laboratory, some additional information is available. Experience has shown that Tag–Heppenstall meters that are in good working order can be readily adjusted by empirical tests to give results in accordance with a specified calibration. Several Universal meters have also been shown to give uniform results, and there is reason to believe that these meters will retain their calibration during a reasonable working life. Discussions with other chemists suggest that some variation in setting may exist among replicates of some other instruments. Accordingly, the calibration equations reported in this paper may not be accurate for all examples of each meter. And moreover, these calibrations apply only with Canadian hard red spring wheat and would not be expected to give the most accurate results for wheats of other classes. Any laboratory purchasing a new meter should check the calibration and recalibrate if this seems necessary.

There is also the question of whether repetition of this study would place the meters in the same rank order of accuracy. The statistical significance of differences between standard errors of estimate cannot be computed from the data for a single study. But experience suggests that it is of the order of 0.3 percentage units or less. On this basis, the Tag–Heppenstall meter yields the most accurate estimates of moisture content and is followed by the Universal meter. The remaining meters fall into four groups listed in order of accuracy as follows: Tag–Dielectric and G.R.L.; Patterson and Steinlite; Mullard and N.P.L.; and Marconi and Toplis–Simpson.

Even the best of these meters will give an erroneous estimate of the moisture content of a considerable proportion of any series of samples. This is to be expected. The meters measure electrical properties of the grain, which are functions of several other properties and conditions of the samples of which moisture content is only one, though the most important. Among these other factors, the distribution of the moisture is known to affect electrical properties. Accordingly, a calibration established for the average distribution of moisture in normal samples is not accurate for samples in which the distribution is abnormal because the grain is freshly harvested, recently tempered, or recently subjected to rapid drying. The packing of the sample in the test

cell also has a major effect on electrical properties, and most meters therefore have loading hoppers designed to reduce this source of variation. For these and other reasons, not yet fully investigated, an accurate prediction of moisture content by electrical methods is not likely to be achieved by any meter for all samples in any random series.

Besides being the most accurate, the Tag–Heppenstall meter provides the most rapid tests, has a uniform temperature correction coefficient, and requires no balance for weighing the sample. These advantages are offset in part by the change in calibration as the rolls wear; though the original calibration can be re-established by simple empirical methods. In addition to its standing of second in accuracy, the Universal meter has the decided advantage of requiring no batteries or external source of current. Among the other meters, several have minor advantages of various kinds, but these are not considered sufficient to offset the greater accuracy of the two resistance types.

Acknowledgments

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